

Influence of liquid nitriding on the properties of the electric steel 10

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Abstract. Liquid nitriding (carbonitriding) effect on hardness, wear resistance and magnetic properties of electric steel grade 10 is reviewed here, as well as on the electric contact resistance. It was found that hot-rolled steel 10 loses its hardness as a result of annealing. Surface carbonitriding of specimens generates an improved layer of higher hardness, high wear resistance and increased electrical resistance. The specimens carbonitrided have contact electric resistance higher than that of hot-rolled specimens after descaling. The magnetic permeability of 'grade 10' steel does not practically change after annealing and carbonitriding, while the magnetic permeability corresponds to the magnetic field strength in a very typical way with typical parameters for carbon steels. Traditional methods of processing electrical steels, carried out in order to achieve such a complex effect, combine heterogeneous processes, such as electroplating, vacuum, plasma, high - temperature annealing, coating-are very expensive and difficult to implement. Carbonitration is more accessible for practical use and has a significantly lower cost. The results obtained can be used in the design of magnetic circuits for various electric devices.

1. Introduction

Electric steels are used for the manufacture of magnetic conductors of electric machines and devices. A significant part of them is technically pure iron with a low hardness of ~HB130 (HV130). After some machining and peening, its grains get refined and some residual stresses are induced, and as a result, the magnetic permeability reduces and the coercive force increases. To improve (restore) electromagnetic properties of electric steels, annealing is practiced [1]. Another special thing about electric steel is dielectric coatings applied on the surface, in order to reduce Foucault currents and electromagnetic losses during operation [2, 3]. In addition, some parts made of electric steel need working surface friction wear protection e.g. electromagnetic clutches. For this purpose, the surface is coated with a precipitated layer of 3 to 5 microns of chemical nickel (in sodium thiophosphate baths) and then boron carbide powder is sprayed. A solid high wear resistant boride and nickel boride (~HV2015) layer is formed during follow-up annealing [4]. This hardening is definitely effective as the hardness is raised more than by 15 times (HV2015/HV130) but is achieved with a complex of heterogeneous processes (galvanic, plasma, vacuum), which makes it expensive and of little feasibility.

Liquid nitriding or carbonitriding can be considered a promising practice that improves the mechanical and electromagnetic properties of electric steel. It is carried out at ~570 °C, which is high enough to eliminate peening hardening, residual stresses and, as a result, improve the electromagnetic



properties. In addition, a hard coating is formed on the surface, which increases the wear resistance and increases the durability of parts [5].

The purpose of this research is influence of liquid nitriding (carbonitriding) on electromagnetic and hardness properties of no-alloyed electric steel grade 10.

2. Material, processing and preparation of specimens

Specimens for this research were cut mechanically from 5mm hot-rolled steel sheets. Chemical composition, weight %: C – 0.08, Si – 0.17, Mn – 0.17, Cr – 0.04, Ni – 0.02, Cu – 0.05, S – 0.006, P – 0.012. Mechanical properties per certificate: tensile strength is 405 MPa, yield strength is 290 MPa, percent elongation is 39.5 %. Electromagnetic and mechanical properties were determined using specimens ‘as received’ and after annealing at 800 °C and 950 °C. Annealing conditions and hardness before and after annealing is shown in Table 1. Then all the specimens were carbonitrided and reexamined. Carbonitriding (liquid nitriding) was performed in an industrial bath with the following sizes: d 450 mm, h 900 mm with molten potassium cyanide Ch (KNCO) at 575 °C.

Table 1. Annealing conditions for steel 10 specimens and their hardness.

Protective annealing environment	Annealing temperature cycle	Hardness, HRB TR-5006 device	
		Before annealing	After annealing
Vacuum: residual pressure (4–8)10×10 ⁻⁴ Pa	heating from 400°C to 800 °C for 1 hour soaking at 800 °C for 4 hours cooling in the furnace from 800 °C to 400 °C, then in the air for 4 hours	90	78
Argon purging inside the furnace, 20 l/min	heating from 400 °C to 950 °C for 2 hours soaking at 950 °C for 3 hours cooling in the furnace from 950 °C to 400 °C, then in the air for 15 hours		62

3. Mechanical tests

The specimens were measured for hardness by the HRB scale before and after annealing with TR-5006 device. It was found (Table 1) that the initial hardness HRB 90 decreased to HRB 78 during annealing at 800 °C, and even more after annealing at 950 °C i. e. to HRB 62. Specimen surface hardness was measured with PMT-3M device after short-term carbonitriding for 10 minutes at various loads on the indenter. The curves of Figure 1 show: the hardness of the steel carbonitrided ‘as received’ was HV102, with low indenter load (10 g), which is slightly higher than HV 77–89 obtained at loads = 20 g. This difference is explained by the fact that the carbonitration layer thickness is related to the indenter penetration depth [4, 5]. Therefore, the values measured represent an integral hardness of the carbonitrided layer and that of the base metal under it, which is not as solid.

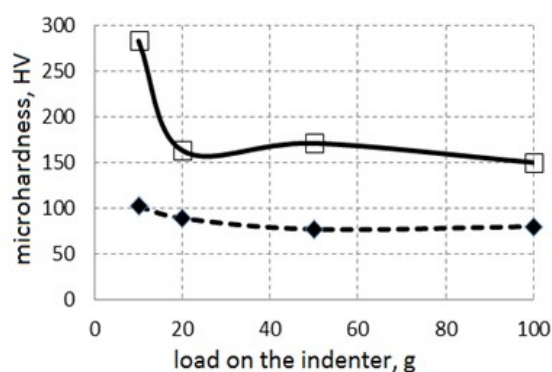


Figure 1. Microhardness of specimen surface
 - -♦- - carbonitrided ‘as received’ (without annealing)
 —□— carbonitriding after annealing at 800°C

It is obvious that the higher the indenter load is, the deeper it penetrates, and the greater is the effect of the solid carbonitrided layer on the resulting hardness. Hence, the hardness value at low indenter load is higher than at higher loads. Pre-annealing at 800 °C caused increased hardness of the surface carbonitrided (Figure 1). It is logical to assume that the diffusion processes were easier for them during carbonitriding, together with increased thickness of the hardened layer. This is also indicated by the hardness HV102 increased to HV283 at a low load of 10 g per indenter.



Figure 2. L-shaped hardness and magnetic permeability specimens.

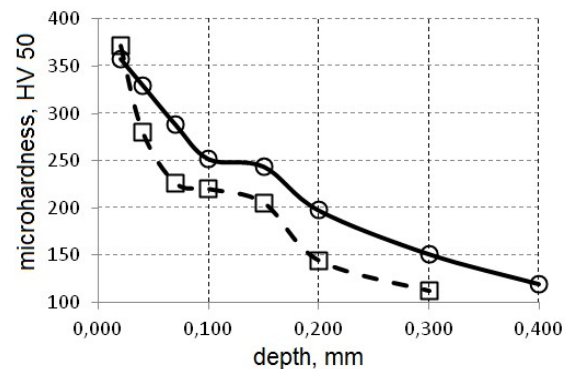


Figure 3. Hardness distribution from the surface after 3 hour carbonitriding
—○— as-received - -□- - annealing 950°C

The steel 10 was tested for magnetic permeability using a magnetic conductor made up of L-shaped specimens shown in figure 2. Some of the specimens were annealed (950 °C) before carbonitriding for 3 hours and some were carbonitrided ‘as received’. Then some samples were cut off from the L-shaped specimen ends designed for this purpose and used to produce cross-section hardness test specimens. See Figure 3 for measurement results.

Figure 3 shows that the cross-section hardness near the surface is ~HV 360. This value is near the value obtained when measuring the surface hardness of a specimen carbonitrided after annealing (Figure 1). L-shaped specimens hardness goes down away from the surface, but it is slightly higher on a specimen without annealing than that of specimens annealed before carbonitriding. This is probably due to the higher hardness of the base metal whereon the thin carbonitrided layer is located (Table 1).

Since the late 90’s of the last century, UZIT-3-type ultrasonic hardness measurement devices have become widespread in the industry. Their determine hardness by the resonant frequency of a magnetostrictive rod with a diamond pyramid indented in the measured surface. Besides, the indentation force is set low at the device, so the mark depth is ~12 microns [6]. This type of device has an advantage over stationary hardness meters that is it can be used for hardness measurement of parts in the production process directly covering up to 100 % of the product output.

In this regard, the hardness of steel 10 was measured after carbonitriding with the UZIT-3 device. It was found that the hardness of specimens annealed at 950 °C is HRC60 after 3 hours of carbonitriding, which is equal to HV~800 by Vickers, and HRC56 (HV~700) for specimens ‘as received’. These values are higher than the hardness readings of the microhardomer shown in Figure 3, and therefore need to be explained.

The area available for measurement was maximized so as to refine the carbonitrided layer hardness values. For this purpose, the surface of the specimen was polished at an angle of ~8 degrees. As a result, the visible sector of the hardened layer on the cut (Figure 4) increased by ~8 degrees, and the indenter had no problem to reach it.

In addition, the indenter load was reduced by half (25 g), which minimized the probability for the indenter to penetrate through the thin solid carbonitride layer to the base metal, which is softer. A FUTURE TECH FM-300 (Japan) microhardness meter was used to make measurements under

control of Thixomet software. The hardness distribution through the carbonitrided layer depth is shown in Figure 5. In addition, the specimen measurement values were multiplied by 0.13. According to the curve, the highest hardness HV₂₅ 800 to 1000 occurs up to 15 microns deep and indicate the carbonitride layer thickness. The underlying diffusive nitrogen saturated zone of the metal 35 microns deep has its hardness stabilized at ~HV₂₅ 530. Thus, it is established that the actual hardness of the thin solid carbonitride layer (15 µm) of on the surface of the steel 10 is HV₂₅ 800 to 1000, and its lower values shown in Figure 3 are explained by the indenter penetration in the softer base metal below.

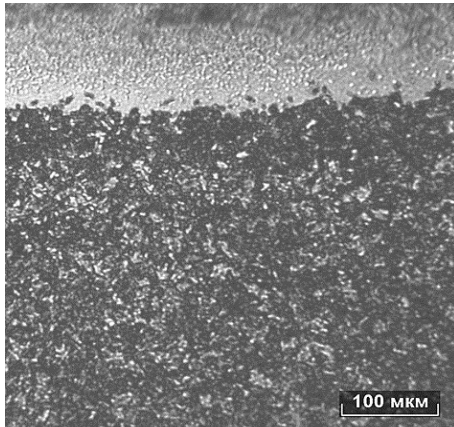


Figure 4. Carbonitrided layer on the metallographic specimen polished at 8° to the surface.

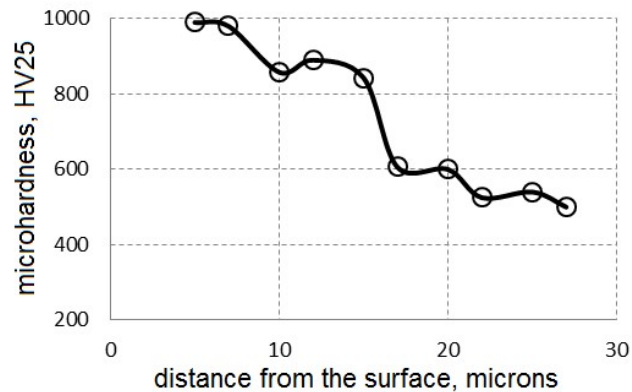


Figure 5. Hardness distributed through the depth of the specimen carbonitrided for 3 hours after annealing at 950 °C.

The carbonitriding effect on the steel 10 wear resistance can be described by the results obtained by dry disk-and-pad friction on the Mi-1M friction machine. It is established that the carbonitrided steel 10 disk with normalized steel 45 pad wears ~10 times lower than the normalized 45 steel disk in the first stage, and ~20 times lower every 5 minutes in the 4th stage of the test.

4. Magnetic properties examination

The following specimens (Figure 1) were made of grade 10 steel in four different ways: 1) hot-rolled; 2) after annealing 950 °C (without carbonitriding); 3) after carbonitriding of hot-rolled specimens; 4) after annealing 950 °C and carbonitriding.

The magnetic steel properties before and after carbonitriding were examined using the ammeter and wattmeter method [8]. The magnetic conductor is made of two L-shaped elements that form a closed loop when matched. Two windings were placed on the magnetic conductor i.e. generator (primary) winding and measuring (secondary) winding. Sinusoidal 50 Hz voltage was applied to the generator winding, and magnetic field strength, magnetic induction and magnetic permeability was determined from the measurements of the primary winding current, secondary winding voltage and active power consumption. Current and voltage were metered with M320 and M890F multimeters; active power consumed was metered with a D539 wattmeter. The supply voltage was adjusted with a laboratory transformer (LATR).

The magnetic induction amplitude value was calculated by the equation from [9], assuming sinusoid nature of the magnetic flow:

$$B_m = \frac{\sqrt{2}E_2}{2\pi fSW_2}, \quad (1)$$

where E_2 – effective EMF value induced in the secondary winding, f – supply voltage frequency, S – magnetic conductor cross sectional area, W_2 – turn number of secondary (test) winding.

When real currents and voltages are replaced with equivalent sinusoidal currents and voltages (equivalence determined by equality of RMS values) and also with account of active magnetic

conductor losses, and provided that winding and magnetic conductor parameters are reduced to the primary winding, finally, the magnetic force amplitude equation will take the following form:

$$H_m = \sqrt{2} \cdot \frac{W_1}{l_{cp}} \cdot \sqrt{I_1^2 - \left(\frac{P_{CT}}{E_2} \frac{W_2}{W_1} \right)^2} \quad (2)$$

where I_1 is a current value in the primary (generator) winding, l_{cp} is an average magnetic conductor length, W_1 and W_2 are winding turn numbers, $P_{CT} = P_1 - I_1^2 R_1$ are magnetic conductor losses, P_1 is active power input, R_1 is active primary windings resistance.

Relative magnetic permeability μ is defined as a ratio of the magnetic induction amplitude to the magnetic force amplitude and magnetic constant: $\mu = B_m / (H_m \mu_0)$.

The magnetic characteristic determining method stated makes possible to adequately evaluate the heat treatment and carbonitriding effect on steel magnetic properties.

Electric properties were measured by ten points with primary winding currents ranging from 0.05 A to 0.3 A, which corresponds to the magnetic force from 250 A/m to 2000 A/m. The primary winding was heating up in the measurements and its active resistance varied from 127 Ohms to 140 Ohms.

The magnetic permeability dependences on the magnetic force turned out to be typical for carbon steels (figures 6, 7) [10, 11]. The representative peak of the curve $\mu(H)$ is observed in the magnetic force range of 300 to 400 A/m. The magnetic conductor gets saturated and the limit magnetic induction values are generated starting from 1000 A/m and higher.

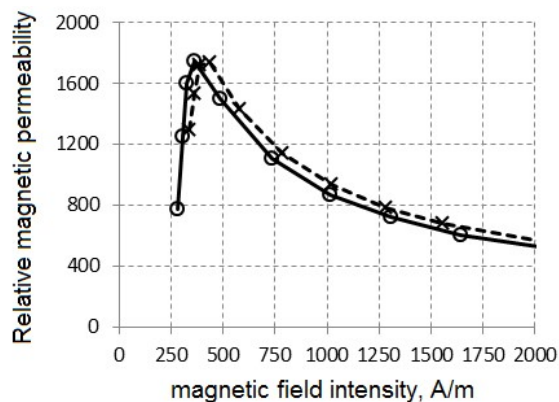


Figure 6. Relative magnetic permeability of specimens without carbonitriding
—○— without annealing (initial state)
---x--- annealing 950 °C.

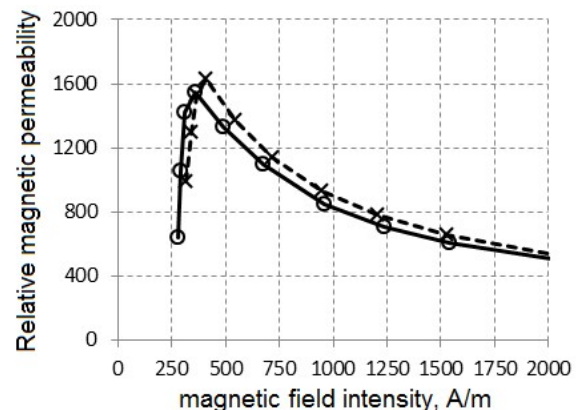


Figure 7. Relative magnetic permeability of specimens after carbonitriding
—○— carbonitriding without annealing
---x--- carbonitriding after annealing 950 °C.

The curves show that the magnetic properties of the steel are practically the same in its initial state, after annealed 950 °C and after carbonitriding; some minor differences are within the tolerance. It can be concluded that 4-hour annealing at 950 °C and carbonitriding at 575 °C have practically no effect on the magnetic properties of the hot-rolled steel 10.

5. Contact resistance examination

A specimen (L-shaped, Figure 1) for contact resistance measurement was placed between the same L-shaped specimens (covers) polished to the shining metal in a hot-rolled state. The measuring device electrodes were pressed against the covers with a force of 30 N. Thus, the measurement pattern used four contact (transient) resistances: 2 between the electrode and the cover and 2 between the cover and the specimen. SEW 6237 DLRO milliohm-meter was used for the measurements. Three series of

experiments were made with the following specimens: 1 is a hot-rolled specimen polished to the shining metal; 2 is a hot-rolled specimen with a surface scale layer; 3 is a carbonitrided specimen without surface polish.

The results achieved show the following: scales on the specimen surface make its contact resistance 59 times higher (1059/18), and a carbonitrided layer make it 3.6 times higher (65/18). The high hot-rolled specimen resistance is explained by a dense oxide film (scale) formed in the heating, combined with high rolling contact pressure. On the contrary, the carbonitrided layer, as it well known [5, 6], has a porous thin (5 microns) structure that with a dielectric property of a chemical carbonitride.

6. Conclusion

- The electrotechnic hot-rolled steel 10 relaxes its hardness from HRB 90 to HRB 62–78 as a result of annealing.
- The steel 10 carbonitrided in the hot-rolled state for 10 minutes generates a hardened layer on the surface with a hardness of 283 HV10. This steel annealed and carbonitrided for 3 hours generates a hardened layer harder than that of these steels in hot rolled condition: HRC 60 and HRC 56, accordingly (UZIT-3 device). The measurements with deeper indenter penetration into the surface (PMT-3M device), give the opposite result (Figure 3), as it is much more influenced by the base metal, which loses the hardness as a result of annealing.
- The solid carbonitrided surface brings a significant (10–20 times) wear reduction to the steel 10 under dry sliding friction on the normalized steel 45.
- Annealing 950 °C and carbonitriding at 575 °C has practically no effect on the hot-rolled steel 10 magnetic properties.
- The contact electrical resistance of carbonitrided specimens is 3.6 times higher compared to the resistance of hot-rolled specimens cleaned from scale. Besides, the porous structure of the carbonitrided surface is better for dielectric varnish coating that has to adhere to it.
- The results obtained can be used in the design of magnetic circuits for various electric devices.

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